



Effects of Artificial Aging Procedure on Flexural Strength of Translucent Zirconia

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ABSTRACT

Objective: The aim of the study is to compare the effect of artificial aging procedure on two types of dental zirconia for flexural strength with two different thickness before and after aging procedure.

Materials and Methods: A total of (80) samples were used in this study; each group contained twenty samples; two groups contained samples with a thickness of 1 mm, and two groups contained samples with a thickness of 1.5 mm; two groups contained samples from ZirCAD MT Multi, while the other two groups contained samples from DD Cube X2 ML. After applying artificial aging by autoclave for five hours at 134⁰ C,0.2 bar to half of each group's samples, the test was conducted by using paired t-test where significant p-value of ($p \leq 0.01$).

Results: The statistical results of flexural strength of the two materials the ZirCad MT Multi (4Y-TZP&5Y-TZP) and the DD Cubic X² ML (5Y-TZP) compare the results before and after aging and the results show that there is no significant difference appear after aging in the two materials.

Conclusions: Increase the thickness of the material result in decrease in flexural strength also result in increased risk for low temperature degradation.

Keywords: zirconia, flexural strength, artificial aging

and toughness which is one of the reasons that the material is often described as the ideal substitute for metal alloys for cores and frameworks in veneered restorations [3][4].

The majority of zirconium oxide ceramics are utilized in refractory applications. At elevated temperatures, they undergo phase changes. At normal temperature, zirconium oxide's stable phase exhibits monoclinic symmetry. It initially changes into a tetragonal phase at temperatures between 1000 and 1100°C, and then into a cubic phase at temperatures over 2000°C. However, these transformations cause significant volume changes: when zirconium oxide is heated, a 5% volume reduction occurs; when zirconium oxide is cooled, a 3% volume gain occurs. This last retransformation into the monoclinic phase is analogous to the martensitic transformation that occurs in steel. [5]

The frequency of framework fracture was shown to be closely linked to the FPD design, with inlay retained FPDs (IRFPD) having the greatest failure rate [6][7].

Certain scientists have raised worry regarding the aging or LTD of transparent zirconia's as a result of the metastable tetragonal phase spontaneously transforming to the monoclinic phase is processed in a humid medium.[8]

The aim of the study is to compare the effect of artificial aging procedure on two types of translucent zirconia for flexural strength of different thickness before and after aging procedure. The null hypotheses planned for the present study was that increase thickness does not affect flexural strength and artificial aging the translucent zirconia.

II. MATERIALS AND MEHODS

The materials were used in this study (Table 1).

I. INTRODUCTION

In dentistry, the increasing demand in esthetic to achieve a life like match prosthetic and craft art, on the other hand, material and technique involved in jewelry making lead to introduction of ceramic. [1]

Zirconia has excellent flexural strength and fracture toughness as a consequence of a physical characteristic called transformation toughening[2]. The unique transformation toughening properties of Y-TZP give the material high mechanical strength



Table 1. The two translucent zirconia with the same color A2.

Material	Groups A	Group B	Manufacture	Origin	Dimension	Patch No
ZirCad MT Multi ^a	1mm	1.5mm	ivoclarvivadent	Swissland	98.5x16 mm	686878
DDCubX ^{2b}	1mm	1.5mm	dental Direkt	Germany	95.5x14 mm	G852001

^aThe new translucent dental zirconia involved increasing the content of Y₂O₃, resulting in two crystalline materials: 4Y-TZP (4 mol% Y₂O₃) and 5Y-PSZ (5 mol% Y₂O₃). Due to the increased Y₂O₃ content, cubic phase occurs alongside metastable tetragonal phase. The quantity of the cubic phase increases from around 25% in 4Y-TZP the main phase (more than 50%) which is why 5Y-TZP is sometimes referred to as partially stabilized zirconia (5Y-PSZ).

^bis based on 5 mol% yttria oxide, which leads to a stabilization of approx. 53% cubic and 47% tetragonal crystals. The DD cubeX² ml give 49% translucency at 1450° sintering

Experimental design

A total sample of 80 standardized translucent zirconium, divided into four groups of 20, were used inflexural strength. The four groups were divided according to different thicknesses of the translucent zirconium, 1 mm and 1.5 mm thickness from ZirCad MT Multi and same number of samples form DD cube X2 (Table 1).

Design and production of translucent zirconium

Prepare 80 specimens of dimensions as specified in table 1 for both types of Translucent zirconium. Samples design in the CAD/CAM, apply the design on the zircons disc to fabricate the sample by using carbide bur inside the machine cut the samples to exact design in the software. The samples that finished from the CAD/CAM machine, we cut the rods that support and hold the sample by carbide bur to obtain the sample, Fire the specimens in sintering furnace (ZIRKONOFEN 600/V2) accordance with the manufacturer's instructions modified as needed due to specimen dimensions. The program 1 in the device for 8 h (first 3 h heating up to 1500° then 2 h hold up at 1500° then cooling down for 3 h). After finished the samples from the sintering, The samples then finishing by use Dia zircon polish (Diatessen) to get polish surface. Check all samples for the dimensions by digital caliber.

Flexural samples

- 1 mm thickness: 13 mm ± (0.02 mm) length x 4 mm (±0.02 mm) width x 1 mm (±0.02 mm) thickness

- 1.5 mm thickness: 18 mm (±0.02 mm) length x 4 mm (±0.02 mm) width x 1.5 mm (±0.02 mm) thickness.

Note: every length of sample is over by 3 mm to prevent the sample from slippage from the base road during applied force according to ISO 6872-2015.

Aging of samples

All samples underwent artificial aging at same time according to ISO 13356-2015. Each sample put inside the glass test tube and put all the tube in the stainless-steel rack. For aging we used autoclave unit (model B, type Tenda, Wesson, China) following ISO 13356 – 2015 recommendations, then test tube rack with sample put inside the autoclave unit under a pressure of 2.1 bars with a temperature of 134° C) for 5h this time give us from 15 to 20 year at patient mouth [9].

Three-point Flexural test

For this test we used universal testing machine (Gester gt k03b) but first we made the base for holding the sample during the test function the base for holding the sample we made the base from the metal material by Torna Machine and the base adjustable to desire space need between holding road, then we holded the sample on the two base road (each road 2.5 mm diameter) and apply the force by metal road head diameter 2.5 mm first on the first samples 1 mm (±0.02 mm) with 13 mm (±0.02 mm) on the middle where is the center to center between base rods was 10 mm and second on 1.5 mm (±0.02 mm) samples with span 18 mm (±0.02 mm) where is the center to center between base rods was 15 mm. Specimens were loaded in a universal testing machine (Gester gt k03b) at a cross head speed of 0.5 mm/min while the base is stable until fracture occurred.

The maximum load of sample fracture was recorded in newton (N) and we need the force to be measure in megapascal (Mpa) so we used formula describe in ISO 6872-2015 as follow:

$$F = 3PI / 2wb^2$$

were

P is the breaking load, in newtons.

l is the test span (centre-to-centre distance between support rollers), in millimeters.

w is the width of the specimen, i.e., the dimension of the side at right angles to the direction.



of the applied load, in millimetres.
 b is the thickness of the specimen, i.e., the dimension of the side parallel to the direction of the applied load, in millimetres.

Statistical analysis

The data obtain were statistically analyzed by spss program by using paired t – test where significant p-value of ($p \leq 0.01$)

III. RESULTS

Results for flexural strength

Flexural strength result for ZirCad MT Multi

Descriptive statistics of flexural strength of ZirCad MT Multi - group A (1mm thickness) and group B (1.5 mm thickness) for the sample before and after aging.

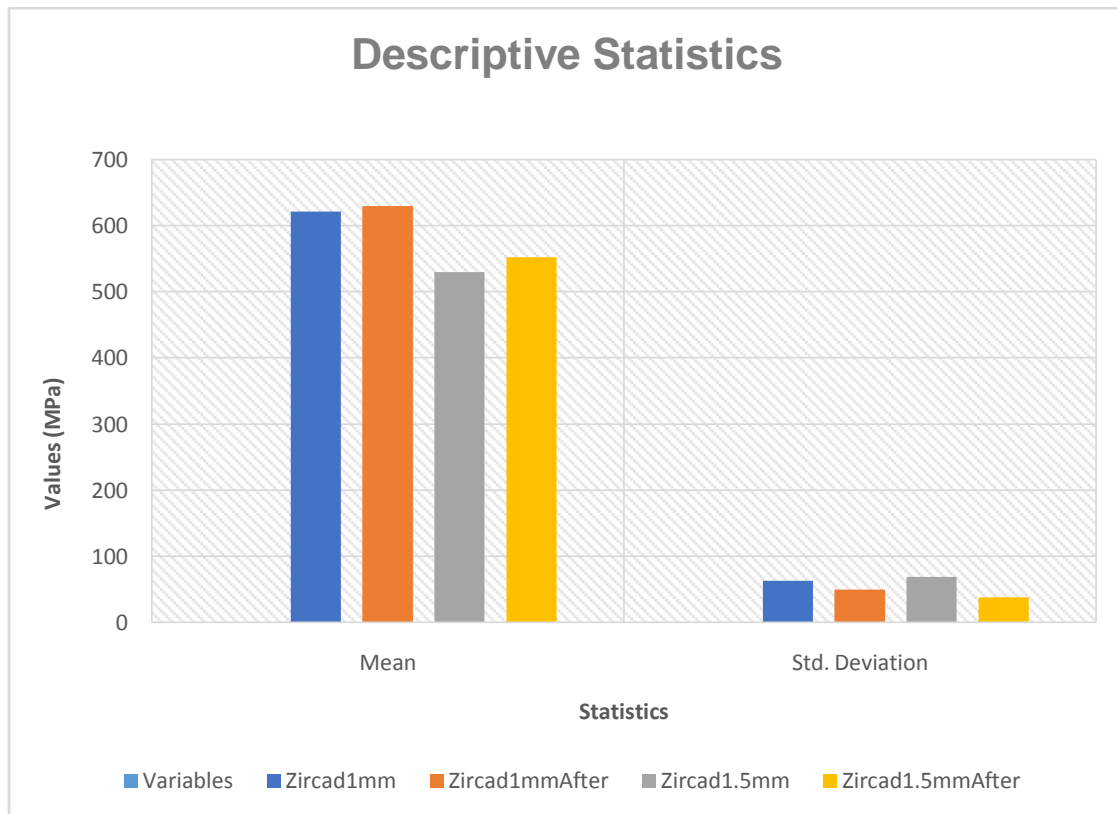


Figure 1: Bar chart show the mean value and stander deviation value of all ZirCad MT Multi before and after aging.

Paired t-test was used statically to test the flexural strength data between the ZirCad MT Multi material before and after aging of the samples and show no significant difference ($P \leq 0.01$), as shown in (Table 2).

Z groups	Mean difference	Std. Error Mean	t	df	Sig (2-tail)
Group A (ZirCad MT Multi 1mm –ZirCad MT Multi 1mm After aging)	-8.73525	28.18897	-.310	9	.764
Group B (Zircad1.5 mm – Zircad1.5 mm After aging)	-22.06528	18.67998	-1.181	8	.271

Table 2: Paired t -test of flexural strength result (MPa) comparison between zircad MT Multi before and after aging



Paired t-test used significant difference was used to compare the mean and stander division between the material in each group before and after aging and the results show that no significant difference ($P \leq 0.01$) in group A between the ZircadMT multi 1 mm thickness before and after aging, also no significant difference ($P \leq 0.01$)

showed in group B between the ZircadMT multi 1.5 mm thickness before and after aging.

Flexural strength result for DD Cubic X²MI

Descriptive statistics OF flexural strength of Cubic X² MI - group B (1mm thickness) and group B (1.5 mm thickness) for the samples before and after aging.

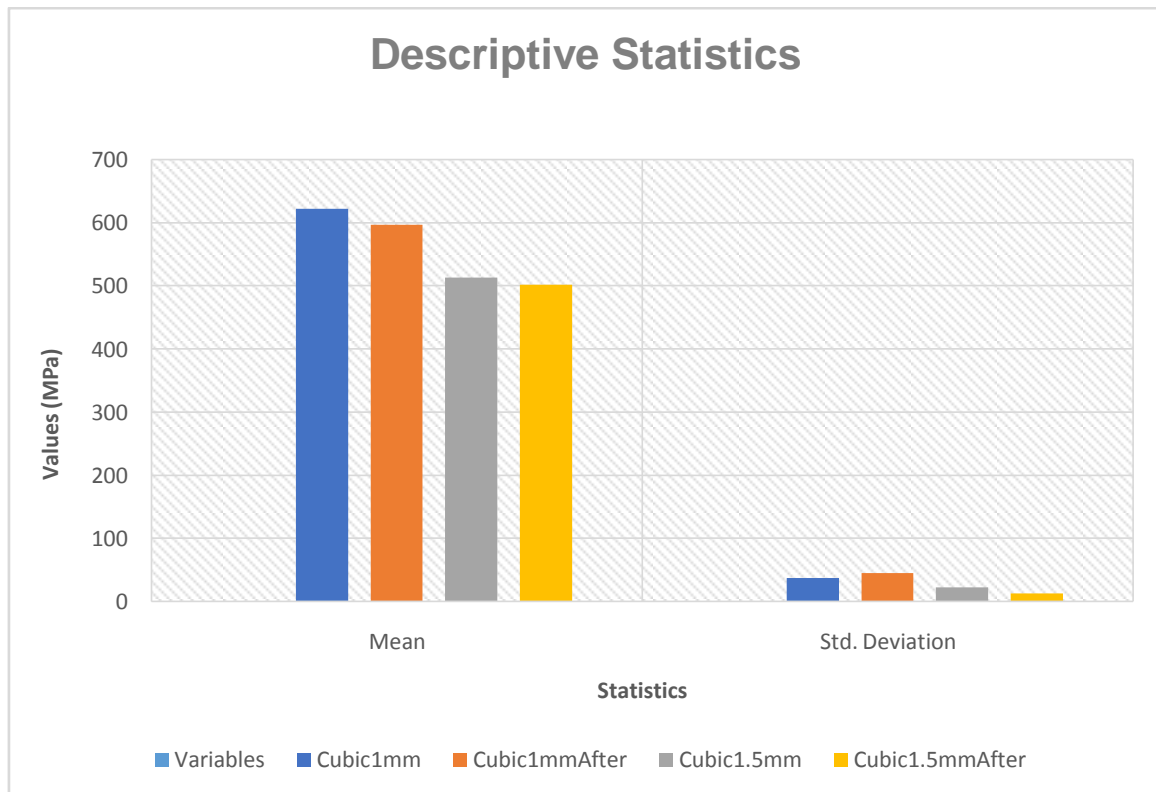


Figure 3: Bar chart show the mean value and stander deviation value of all DD Cubic X² MI before and after aging.

Paired t-test was used statically to test the flexural strength data between the DD Cubic X²MI material before and after aging of the samples and show no significant difference ($P \leq 0.01$), as shown in (Table 3).

Z groups	Mean difference	Std. Error Mean	t	df	Sig (2-tail)
Group A (Cubic 1mm - Cubic 1mm After aging)	25.85639	25.77564	1.003	8	.345
Group B (Cubic 1.5 mm - Cubic 1.5 mm After aging)	10.70275	6.35852	1.683	9	.127

Table 3: Paired t -test of flexural strength result (MPa) comparison between DD Cubic X² ML before and after aging



Paired t-test used significant difference was used to compare the mean and standard deviation between the material in each group before and after aging and the result show that no significant difference ($P \leq 0.01$) in group A between the DD Cubic X2 MI 1 mm thickness before and after aging, also no significant difference ($P \leq 0.01$) showed in group B between the DD Cubic X2 MI 1.5 mm thickness before and after aging.

IV. DISCUSSION

In present study, we use Yttria full stabilized tetragonal zirconia polycrystal (Y-TZP) because most material use now a day in fabrication of prosthetic part of teeth because of its mechanical and optical properties, as list before in the literature review there are different type of (Y-TZP), we use in our study two type of material the 5Y-TZP and other material contain (4Y-TZP & 5Y-TZP) in each disk, and apply to the materials artificial aging by autoclave and after that evaluate the flexural strength (mechanical properties) of the material at two thickness before and after aging and compare between the two material.

The statistical results of flexural strength of the two materials the ZirCad MT Multi (4Y-TZP & 5Y-TZP) and the DD Cubic X² ML (5Y-TZP) compare the results before and after aging and the results show that there is no significant difference appear after aging in the two materials as shown in table (2 and 3).

The mean of the ZirCad MT Multi in the two thickness have shown slight increasing after aging but statistically not significant while the mean of DD cube X²ML has shown slight decreasing but statistically not significant. Where in the non-aged sample the result show that the ZirCad MT Multi has higher result in 1mm thickness was 688.5 and in 1.5 mm was 602.4 and lower value for 1 mm thickness was 477 and in 1.5 mm was 391.6, while the non-aged value for the DD cubic X² ml showed that the higher result in 1mm thickness was 702 and in 1.5 mm was 540.5 and lower value for 1 mm thickness was 572.6 and in 1.5 mm was 492.7.

The aged sample the result showed that the ZirCad MT Multi the higher result in 1mm thickness was 707.8 and in 1.5 mm was 623.3 and lower value for 1 mm thickness was 565.3 and in 1.5 mm was 485.3, while the non-aged value for the DD cubic x2 ml showed that the higher result in 1mm thickness was 688.5 and in 1.5 mm was 522.2 and lower value for 1 mm thickness was 559.8 and in 1.5 mm was 485.3. The null hypothesis that increase thickness does not affect flexural

strength and artificial aging the translucent zirconia was rejected.

Flexural strength of these study before and after aging conform with the results of those **Stawarczyk et al., (2016)** who state that there is no significant difference between the non-aged and aged group at ($p < 0.05$). when using aging by autoclave for 5 h at 134 C for Zenostar (ZS), DD Bio ZX2 (DD), Ceramill Zolid (CZ), InCoris TZI (IC) and Ceramill ZI (CZI), the mean and standard deviation (std) show that the flexural strength decreased after aging just for the ZS and increased for the other. [10] **Flinn et al., (2016)** who stated that there is no significant difference between the non-aged and aged for monolithic zirconia group for two type of commercial material Katana HT13 and Katana ML but there is significant difference for other two material Prettau and BruxZir and this was confirmed with our study. [11] **Camposilvan et al., (2018)** who stated that there is no significant difference between the non-aged and aged group when using aging by autoclave for 6 h, 18 h at 134 °C, 2 bar for full-strength 3Y-TZP grade “Aadva ST”, improved translucency “Aadva EI”, highly translucent and partially cubic “Aadva NT” and highly translucent, partially cubic and multi-layered inchroma “Katana UTML”, the mean and standard deviation (std) show that the flexural strength slight increasing after aging for the different type of Y-TZP and different composition the monolithic, tetragonal and cubic phase and this was confirmed with our study. [12]

Al ghazzawi and Janowski. (2015) disagree with our study, when he made aging for different zircon material some of them was significant change at ($P < 0.05$) when he tested the material for flexural strength (3-point bending), when he compare mean and standard deviation to specimens that apply steam autoclave aging (134 °C under a pressure of 0.2 MPa) for 50 hours for six zircon brands Lava Plus High Translucency (Lav), Argen HT (Arg), ZirLux (Zir), BruxZir (Bru), ZenoStar (Zen), and DDBioZX2 (DDB) and his result show significant difference to all brands except BruxZir (Bru). [13] **Munoz et al., (2017)** also his study disagrees with our study in high translucent zirconia when he tested the material for biaxial flexural strength after he applied the aging protocol by autoclave (134 °C under pressure of 0.2 MPa) for 8 hours and the results showed significant difference ($P < 0.05$) high translucent zirconia brand Prettau Anterior, and the mean of the material show significantly decrease in value after aging. [14] **Prado et al., (2017)** who state that there is significant difference between non-aged group and aged group when he use aging by



autoclave for 20 h at 134⁰ C, 0.2 bar for Zirlux FC zircon and after that check the flexural strength (biaxial test) and this disagree with our study because the flexural strength mean increase significantly ($P < 0.05$) after aging.[15]

In this study documented that the flexural strength decreased after aging but not significant as a result of 3 mechanisms:

- Water reacting with yttria to form yttrium hydroxide, which consumes enough stabilizing oxide to induce conversion to the monoclinic phase.[16]
- Water attack on zirconium-oxygen bonds results in the accumulation of stress due to the movement of the hydroxide ion into the crystal structure (this motion generates defects that act as nucleating sites for subsequent transformation from the tetragonal-to-monoclinic phase). [17]
- Oxygen ion (not hydroxide ion) fills oxygen vacancies created by water isolation.[18]

In our study there is decrease in flexural strength but not significant as appear in DD Cubic X² ml and this also may be related to the absence of transformation toughening in cubic zirconia and the coarser microstructure, the mechanical properties deteriorate significantly.[12]The difference between the DD Cubic X² ML and the ZirCad MT Multi is that the Cubic X²ML contain more cubic phase while the ZirCad MT Multi contain more tetragonal phase, that tetragonal zirconia is frequently supersaturated with alumina to improve its aging properties and reinforce grain boundaries [19].

It would be interesting to study different thickness (less thick than 1mm samples which we used in our study) in combination of a 0.3mm micro veneering layer of porcelain in future research projects.

V. CONCLUSION

Increase the thickness of the material result in decrease in flexural strength for the two materials, also result in increased risk for low temperature degradation, The flexural strength of combination of 4 Y-TZP & 5 Y-TZP show slight increase in flexural strength after aging while the 5 Y-TZP show slight decrease in flexural strength but the two were not significant so it better to use the 4&5 Y-TZP for bridge.

REFERENCES

- [1]. Contrepolis, M., Soenen, A., Bartala, M., Laviolle, O. (2013). Marginal adaptation of ceramic crowns: a systematic review. *J Prosthet Dent*. 110, 447–454.
- [2]. Peláez J, Cogolludo PG, Serrano B, Lozano JF and Suárez MJ. (2012). A prospective evaluation of zirconia posterior fixed dental prostheses: three-year clinical results. *J Prosthet Dent*, 107:373-379
- [3]. Heintze SD, Rousson V. (2010) Survival of zirconia- and metal-supported fixed dental prostheses: a systematic review. *Int J Prosthodont*. 23:493–502.
- [4]. Lughi V, Sergo V. (2010) Low temperature degradation -aging- of zirconia: a critical review of the relevant aspects in dentistry. *Dent Mater J*. 2010; 26:807–820.
- [5]. Christel P, Meunier A, Heller M, Torre J, Peille C. (1989) Mechanical properties and short-term in vivo evaluation of yttrium-oxidepartially- stabilized zirconia. *J Biomed Mater Res*. 23: 45–61.
- [6]. Saridag S, Tak O and Alniacik G. (2013). Basic properties and types of zirconia: An overview. *World J Stomatol*, 2(3): 40-47.
- [7]. Al amlehB, Lyons K and Swain M. (2010). Clinical trials in zirconia: a systematic review. *Journal of Oral Rehabilitation*, 37; 641–652
- [8]. Tong H, Tanaka CB, Kaizer MR and Zhang Y. (2016). Characterization of three commercial Y-TZP ceramics produced for their high-translucency, high-strength and high-surface area. *Ceram Int*. 42:1077-85.
- [9]. Kou W, Garbriellsson K, Borhani A, Carlborg M and Molin ThorénM. (2019). The effects of artificial aging on high translucent zirconia. *BiomaterInvestig Dent*, 6(1):54-60.
- [10]. Stawarczyk B, Frevert K, Ender A, Roos M, Sener B and Wimmer T. (2016). Comparison of four monolithic zirconia materials with conventional ones: contrast ratio, grain size, four-point flexural strength and two-body wear. *J Mech Behav Biomed Mater*, 59:128–38.
- [11]. Flinn BD, Raigrodski AJ, Mancl LA, Toivola R and Kuykendall T. (2017). Influence of aging on flexural strength of translucent zirconia for monolithic restorations. *J Prosthet Dent*, 117:303–9.
- [12]. Composilvan E, Leone R, Gremillard L, Sorrentino R, Zarone F and Ferrari M. (2018). Aging resistance, mechanical properties and translucency of different yttria-stabilized zirconia ceramics for monolithic dental crown applications. *DentMater*, 34:879–90.
- [13]. Al ghazzawi TF and JanowskiGM. (2015). Correlation of flexural strength of coupons



- versus strength of crowns fabricated with different zirconia materials with and without aging. *J Am Dent Assoc*, 146:904–12.
- [14]. Munoz EM, Longhini D, Antonio SG and AdaboGL. (2017). The effects of mechanical and hydrothermal aging on microstructure and biaxial flexural strength of an anterior and a posterior monolithic zirconia. *J Dent*, 63:94–102.
- [15]. Prado RD, Pereira GKR, Bottino MA and MeloRMVL. (2017). Effect of ceramic thickness, grinding, and aging on the mechanical behavior of a polycrystalline zirconia. *Braz Oral Res*, 31, e82:1–9.
- [16]. Lange FF, Dunlop GL and Davis BI. (1986). Degradation during ageing of transformation toughened ZrO₂-Y₂O₃ materials at 250°C. *J Am Ceram Soc*, 69(3):237-240
- [17]. Yoshimura M, Noma T, Kawabata K and Somiya S.(1987). Role of water on the degradation process of Y-TZP. *J Mater Sci Lett* 6:465–7.
- [18]. Chevalier J, Gremillard L, Virkar AV and Clarke DR.(2009). The tetragonal–monoclinic transformation in zirconia: lessons learned and future trends. *J Am Ceram Soc*, 92:1901–20.
- [19]. Zhang F, Inokoshi M, Batuk M, Hadermann J, Naert I and VanMeerbeekB. (2016). Strength, toughness and aging stability of highly-translucent Y-TZP ceramics for dental restorations. *Dent Mater*,32: e327-37.